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# FEASIBILITY STUDY OF COLUMBIUM ALLOY CASTINGS

JANUARY 1971

R.J. BEAUREGARD and J.J. WALTERS  
Avco Lycoming Division  
Stratford, Connecticut 06497

FOURTH PROGRESS REPORT - CONTRACT DMR-64-05-0103  
COVERING 1 MAY THROUGH 30 OCTOBER 1970

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Prepared for

ARMY MATERIALS AND MECHANICS RESEARCH CENTER  
Watertown, Massachusetts 02177

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Fourth Progress Report - Contract DAAG46-69-C-0163

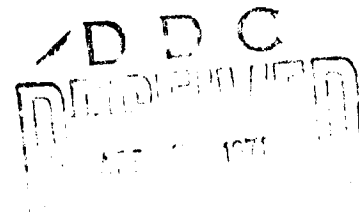
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MANUFACTURING METHODS AND TECHNOLOGY PROJECT

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### ABSTRACT

The purpose of this program is to determine the feasibility of investment-casting columbium alloys. Due to delays encountered by the casting vendor, the program has been extended with a new completion date of 1 March 1971, and this fourth progress report is being distributed to more effectively disseminate the information gained to date. The current reporting period encompasses a length of time equal to two quarters, six months, ending with October 1970.

Thirty-four castings are scheduled to be produced, using consumable-electrode arc-melting and tungsten interfaced molds to prevent mold-metal reaction. Phase I of this program concerns the optimization of mold design and preheat temperature. Phase II envisages the production of sound test specimens, followed by coating, and an environmental and mechanical testing effort.

During this reporting period, three attempts were made to evaluate the effect of venting on porosity in fluidity molds. Successful evaluation was not possible due to poor fluidity in two of the castings and a cleaning accident with the third casting.

Gating techniques for test specimens were evaluated on two C-3015 castings with considerable insight gained prior to the forthcoming final gating selection.

## FOREWORD

This technical report is the fourth progress report submitted in compliance with Army Contract DAAG46-69-C-0163, and covers the scope of work accomplished during the period of 1 May through 31 October, 1970. Proposal 2190.6.1 implements the materials manufacturing project authorized by the contract.

The research and development task dealt with in this report was initiated between the Army Materials and Mechanics Research Center and Avco Lycoming Division to study the feasibility of producing columbium alloy parts by investment-casting techniques.

R. J. Beauregard and J. J. Walters are Development and Project Engineer, respectively, for this study. B. A. Ewing, Chief of Metal and Coating Subsection, has overall responsibility for project management. The study program is under the direction of W. R. Freeman, Jr., Director of the Avco Lycoming Materials Laboratories. This contract is technically supervised by K. D. Holmes of the Army Materials and Mechanics Research Center.

This project has been accomplished as part of the U. S. Army Manufacturing and Technology Program, which has as its objective the timely establishment of manufacturing processes, techniques or equipment to insure the efficient production of current or future defense programs.

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## I. INTRODUCTION

One of the most effective means of increasing the overall performance of a gas turbine engine is to increase the turbine inlet temperature. The limiting factor in the adoption of higher turbine inlet temperatures has been the high-temperature materials capability of the hot-end components. At present, cobalt and nickel-base alloys in both wrought and cast forms are being used extensively in such applications. Although protective aluminide coatings have extended the high-temperature capabilities of these alloys, their low creep-rupture strength and the microstructural instability of the required coating systems generally limit their usefulness at temperatures above 1800 F.

If higher turbine inlet temperatures are to be used, two approaches for upgrading hot-end component life are available: (1) the use of cooling processes, and (2) the development and use of materials with higher-temperature capability. The cooling approach detracts from overall engine performance owing to the use of compressor air. The materials approach has periodically focused considerable attention on the refractory metals: columbium, molybdenum, tantalum, chromium, and tungsten. These metals possess mechanical properties generally acceptable for high-temperature turbine engine usage, although their susceptibility to oxidation generally imposes severe limitations on their utility in air-breathing turbines. Of these refractory metals, the more advanced, low-density alloy systems of columbium rank it as a very attractive candidate for potential turbine applications. To date, considerable effort has been expended by gas turbine manufacturers and refractory-metal producers in an attempt to develop a columbium alloy capable of withstanding temperatures in excess of 2500 F. The principal disadvantage with columbium, however, is its poor oxidation resistance, which necessitates a self-healing protective coating system for satisfactory gas turbine engine operation.

Because of this oxidation problem, past attempts at introducing columbium alloys in high-temperature gas turbine applications have been unsuccessful. Recently, however, interest has been revived in introducing columbium alloys into advanced engine designs. Encouragement grows from improvements in protective coating systems culminating in an Air Force engine evaluation of wrought columbium alloy vanes and the reported development of alloy substrates exhibiting improved oxidation resistance.

In conjunction with recent improvements in protective coating systems and columbium alloy development, technology in the precision casting of reactive and refractory materials has also advanced, holding the promise for improved economy and design efficiency. In particular, recent product improvement work with investment cast titanium, performed by Avco Lycoming<sup>1</sup> at the request of the Army Materials and Mechanics Research Center, has demonstrated that complex configurations can be cast to finished dimensions with virtually no surface reaction with either the molding material or surface environment. It is significant that extremely reactive materials with high melting points, such as titanium alloys, can be investment cast, particularly when the concept is extended to refractory materials that are known to share a common degree of casting complexity.

In view of the combined technological advances recently made with respect to protective coating systems for columbium alloys, oxidation-resistant columbium alloy development, and reactive-metal casting techniques, thought has been advanced toward the casting of columbium into airfoil shapes. The successful implementation and integration of these latest developments into gas turbine hot-section design could well result in a quantum jump forward in gas turbine capability and efficiency. It is the purpose of this program to determine the feasibility of producing investment cast B-66 and WC-3015 columbium alloy vanes with oxidation and mechanical property potential consistent with U. S. Army-Avco Lycoming advanced engine requirements.

## II. TECHNICAL APPROACH

### PROGRAM PLAN

The program to establish the feasibility of casting columbium alloys consists of two phases. In Phase I, mold processing and mold preheat temperature variations will be investigated and optimized using fluidity molds. The fluidity mold, illustrated in Figure 1, consists of a series of "wings" extending from a tapered sprue. Wing cross section increases as the sprue tapers; two wings have 0.030-inch sections, two have 0.061-inch sections, and four have 0.080-inch sections. The degree of fill

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<sup>1</sup>U. S. Army/Avco Lycoming Product Support and Component Improvement Programs, Improved Compressor Project 1111, CY 1968 Quarterly and Final Reports Nos. 3162.1, .2, .3, and .4 and CY 1969 Quarterly and Final Reports Nos. 4212.2, .3, and .4.



obtained in the various wing sections gives a measure of the alloy fluidity obtained under the particular pouring conditions. The optimized conditions determined in Phase I will be applied in Phase II for the preparation of stress-rupture and tensile test bars, thermal fatigue and oxidation test paddles, and cored turbine nozzle vanes for the evaluation of mechanical properties, oxidation resistance and low-cycle thermal fatigue resistance.

During the first three quarters of this program, investigations conducted by Avco Lycoming established the feasibility of investment casting columbium alloys B-66 and C-3015 with no detectable contamination. All casting operations were conducted by REM Metals Corporation, Albany, Oregon, using consumable-electrode arc-melting of the columbium alloys into REM tungsten-interfaced molds.

The optimization of the two major casting variables, mold preheat temperature and number of tungsten interface layers, was also completed. The selection of two tungsten layers and a mold preheat temperature of 1400 F (REM's maximum capability) was considered optimum on the basis of fluidity and freedom from contamination.

During this past reporting period, an attempt was made to eliminate suspected porosity in fluidity molds, using the venting arrangement seen in Figure 2. Gating techniques to be used for test specimen production also were investigated during this time. All molds used for gating investigations were made with two tungsten interface layers and all casting was conducted with molds preheated to 1400 F.

#### PERIODIC ACTIVITY

##### B-66 Alloy

Due to equipment breakdowns, prior production commitments and mold breakage problems encountered at REM Metals Corporation, only one B-66 casting was produced during this reporting period. This one casting, produced from the vented fluidity mold shown in Figure 2, was made on the third attempt after the first two attempts were unsuccessful due to mold breakage during handling prior to pouring. Two attempts to produce a combination test specimen casting for evaluation of gating techniques, using a mold configuration such as shown in Figure 3, were also unsuccessful due to mold breakage before pouring.

### C-3015 Alloy

Four C-3015 castings, consisting of two vented fluidity castings (Figure 2) and two combination test specimen castings, were received from REM Metals Corporation. The first vented fluidity casting fractured in the cleaning solution, necessitating the pouring of a second casting. Each fluidity casting was poured on the second attempt due to mold breakage on the first attempt. The first combination test specimen mold was poured using the gating arrangement shown in Figure 3. The second pour incorporated a modified gating arrangement similar to that shown in Figure 3, but with enlarged top and bottom gates on both the bars and the paddles. The paddles also were center gated (leading edge) on this pour.

### Visual and X-ray Examination

Following casting and cleaning, the vented fluidity castings produced during this reporting period, except the one that fractured during cleaning, were given the same inspection as the previous non-vented fluidity castings, beginning with their photographic documentation for an overall comparison of quality. The wings were subsequently removed, rephotographed and x-ray radiographed. The first C-3015 combination test specimen casting was examined in a similar manner, while the second one was not considered suitable for examination due to excessive mold degassing, which will be discussed later.

### Microstructural Examination

Light microscopy studies were performed on both sprue and wing sections of the fluidity castings and on the test specimens of the combination castings. The preparation of the metallographic specimens followed procedures similar to those used during the preceding quarters. The samples were mounted in diallyl phthalate and ground through 600-grit silicon carbide paper, followed by polishing for 12 to 24 hours on a vibratory polisher using 0.05-micron alumina on a low nap, synthetic cloth.

Following polishing, the C-3015 samples were anodized in a solution of 10 cc lactic acid, 5 cc phosphoric acid, 2 grams citric acid, 20 cc glycerine, 60 cc methanol, and 35 cc distilled water by applying a potential of 60 volts between the sample and a stainless steel beaker (cathode). The B-66 samples were immersion etched in a solution consisting of 20 cc hydrofluoric acid, 14 cc sulphuric acid, 5 cc nitric acid and 50 cc distilled water.

### III. DISCUSSION OF RESULTS

#### CASTING EFFORT

Mold breakage and casting equipment breakdowns have continued to plague the casting effort of this program and have necessitated a five month time extension of the original program. The mold breakage problem is considered quite serious. At present, about 50 percent of the molds are breaking prior to casting. The strength of the REM mold used for columbium castings is apparently insufficient to withstand normal handling. Several attempts have been made to reduce the quantity of broken molds, including changing the zirconia backup layers to alumina, reinforcing the mold with stainless steel wire, and using the higher strength REM titanium mold binder in the zirconia backup layers. All attempts have failed and it appears that the program will continue to be plagued by mold breakage. Further development work will have to be undertaken to develop a non-reactive mold system with superior strength if cast columbium is to become a production reality.

#### CASTING EVALUATION

##### B-66 Alloy

The overall appearance of the one B-66 alloy casting produced this quarter, a vented fluidity specimen, is shown in Figure 4. The amount of fill on this casting (Figure 5) was considerably lower than on previous pours, which precluded the planned analysis of the effect of venting on porosity.

Routine metallographic examination on this B-66 casting revealed the occurrence of the abnormal microstructure shown in Figure 6. The area marked B in the photomicrograph appeared to be an immiscible second phase within the B-66 matrix (area A). Subsequent electron microprobe analysis showed the areas marked B to be rich in chromium, nickel and iron. Since melt contamination was suspected, a wet chemical analysis of the casting was performed. The results, presented in Table I, show that the melt indeed was contaminated. Subtracting the nominal B-66 composition from the analysis, it appears that the melt was contaminated with a 300-series stainless steel. Subsequent spectrographic

analysis of the remaining B-66 at REM revealed the presence of pieces of Permalloy (Ni-16.7 Fe, 4 Mo, .3 Mn) and tantalum. This material mix-up appeared to be the fault of the vendor: Fansteel, Inc. The poor fluidity obtained in this casting may be explained by the result of this material mix-up since, in essence, a different alloy was cast.

#### C-3015 Alloy

The overall view of the first C-3015 vented fluidity casting poured in this period is shown in Figure 7. This casting fractured during the caustic cleaning procedure employed after mechanical knock-out. The caustic cleaning, done in a proprietary solution of hot potassium and sodium hydroxides, is necessary to completely remove the strongly adhering tungsten mold material. The amount of fill on the casting appeared to be similar to previous pours, with two tungsten layers and a mold preheat of 1400 F, although no photographs were taken prior to cleaning. Microstructural examination of a piece of the casting (Figure 8) showed severe internal transgranular cracking. It is not readily apparent, however, if the cracking was a result of residual stresses in the casting or was caused by improper cleaning procedures. Analysis of the circumstances surrounding the cleaning operation led to the suspicion that the cleaning operation was at fault since the problem had not occurred on previous pours and REM had used a smaller cleaning tank with a different caustic solution in an attempt to improve the rate of tungsten removal.

The overall appearance of the second C-3015 fluidity casting is shown in Figure 9. The degree of fill obtained (Figure 10) was, as in the B-66 pour, much lower than obtained in previous pours under the same pouring conditions (two tungsten layers and 1400 F mold preheat) again precluding the planned investigation of the effect of venting on porosity. Discussion with REM indicated that the energy input during the melting of the electrode was less than desirable, leading to the suspicion that the molten metal temperature was lower than on previous pours, resulting in decreased fluidity. On future pours, more care will be taken by REM to insure that the energy input is adequate and consistent.

TABLE I. CHEMICAL COMPOSITION OF B-66 VENTED  
FLUIDITY CASTING

Element	Actual Weight Percent	Nominal B-66 Composition
V	4.5	5.0
Mo	4.95	5.0
Fe	4.5	-
Cr	3.45	-
Ni	3.60	-
Zr	0.93	1.0
Mn	0.18	-
Ti	0.11	-
Si	Trace	-
Cb (Nb)	Balance	Balance

Shown in Figure 11 is the overall view of the first C-3015 combination test specimen casting which used the gating techniques shown in Figure 3. The visual and x-ray radiograph views of the individual specimens are presented in Figure 12. It should be noted that the fractured tensile bar and thermal fatigue paddle were broken during the cutoff operation and mold knock-out, respectively, which suggests that the C-3015 alloy has low toughness in the as-cast condition. The test bars, which had machined gauge sections, are shown in Figure 12 to be x-ray sound and exhibit excellent fill in the gauge length. The thermal fatigue paddles were insufficiently filled and had shrinkage problems. The solid turbine vane was completely filled, although subsequent caustic cleaning severely attacked the trailing edge as shown in Figure 12. Better hand cleaning of the castings will be needed to reduce the time in the caustic cleaning and the consequent attack on the trailing edge. No indications of surface contamination were seen in the microstructures of the vane, the bars, or the paddles.

Figure 13 shows the overall view of the second C-3015 combination test specimen casting that employed the REM titanium mold binder in the zirconia backup layers in an attempt to provide a higher strength mold. The binder appeared to have broken down under the heat of the columbium melt, however, causing severe outgassing of the mold. This resulted in a well sintered tungsten layer on the casting which could not be removed during caustic cleaning (Figure 14). In addition, the breakdown of the binder appeared to release oxygen which subsequently reacted with the surface of the casting. This was detected by electron microprobe analysis, which showed the zone marked A in Figure 14 to be rich in hafnium, columbium, zirconium, and oxygen, and lean in tungsten. In short, the titanium mold binder was unsuccessful for use in the casting of columbium.

This second C-3015 combination test specimen (Figure 13) casting was made using the modified gating technique previously discussed. With respect to the fluidity obtained with this modified gating, the analysis was somewhat complicated by the outgassing of the mold material. Good fill apparently was obtained on all specimens with the exception of one paddle. An unknown factor however, is the effect of a possible gas layer (from the outgassing) on resultant fluidity.

#### IV. FUTURE WORK

The development of gating techniques for the production of cast test specimens is nearing completion. Two additional test specimen molds remain to be poured; one in B-66 and one in C-3015. Due to the mold breakage problem, four molds will be prepared to insure the successful pouring of the two castings. These molds will utilize the normal mold binder with two tungsten layers and a preheat temperature of 1400 F. Two mold configurations will be used, as shown in Figure 15. Several differences can be observed between the gating on these molds and that of the mold shown in Figure 3. First, the gate leading to the turbine vane has been tapered, while the test bars have enlarged end-gates. Paddle orientation and gating size have been modified to explore their influence on castability.

To evaluate these mold configurations, one alloy will be poured into mold A and the other alloy will be poured into mold B. Since previous third quarter work showed the fluidity of both alloys to be similar, it is felt that an accurate appraisal of both gating techniques will be possible with this approach with a simultaneous saving of time.

After analysis of these two castings it is hoped that the gating will be finalized for the production of the remaining thirteen program pours. The only unknown factor will be whether the gating used for solid turbine vanes (to be used on these two pours) will work effectively on cored turbine vanes. Since development of cores at REM for columbium casting has not yet been completed, it has not been possible to use cored turbine vanes in the gating evaluation segment of this program. Completion of core development at REM is expected soon, however, which will allow gating adjustments to be made early in the production of test specimens.

Following completion of the casting effort, acceptable specimens will be coated by Sylvania and Vac Hyd, using their slurry silicide coatings. After coating, the Phase II testing activity will be initiated. Testing in this phase is to include 70 F and 1400 F tensile and 2200 F stress rupture tests of the bars and a combined thermal fatigue and oxidation test to 2200 F maximum temperature on as-coated and ballistically-impacted test paddles.

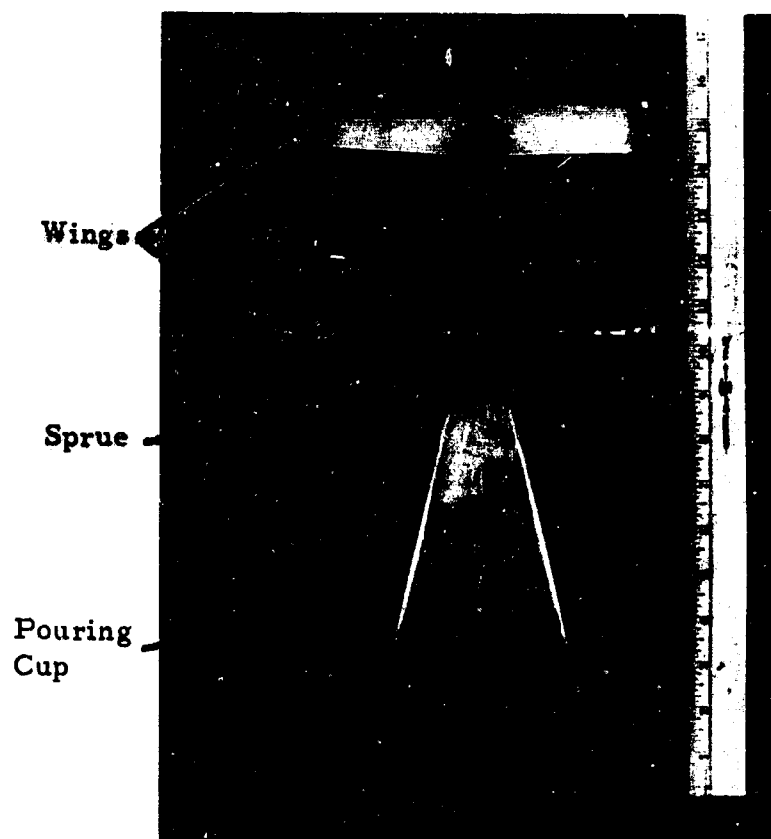
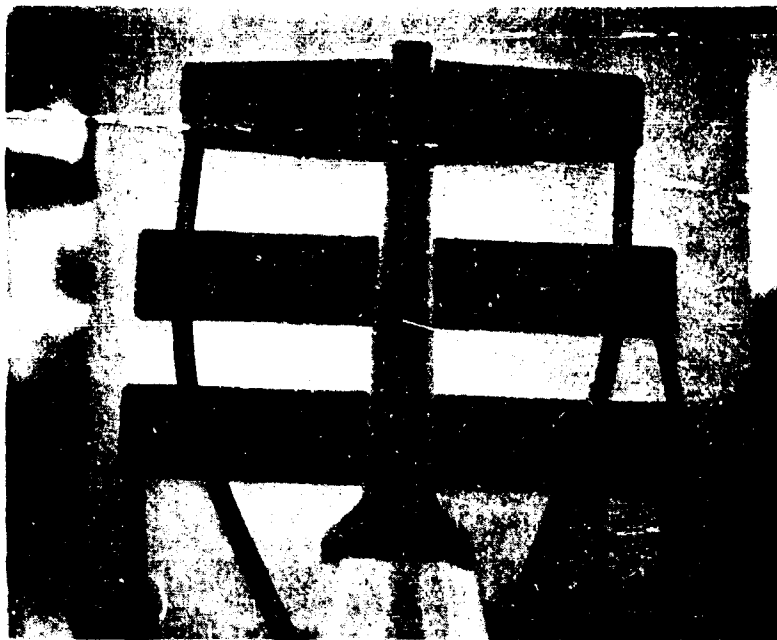


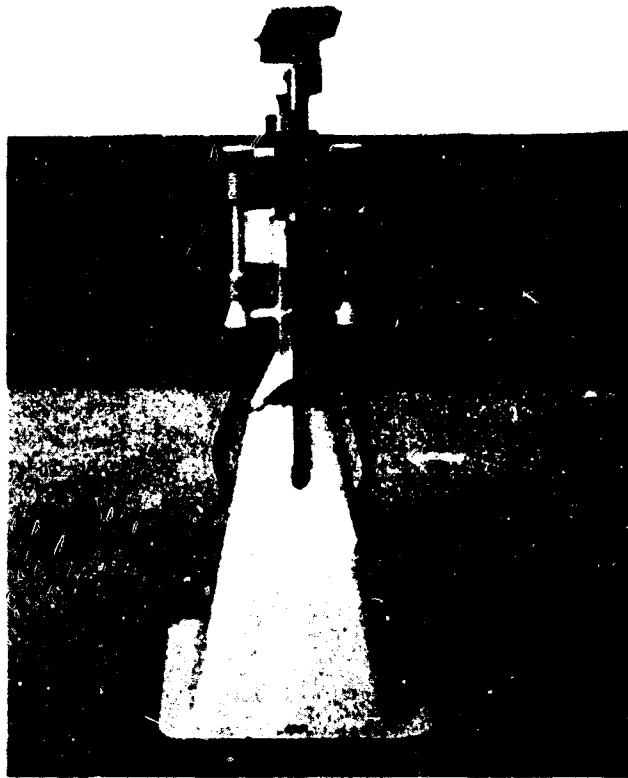
Figure 1. Typical Fluidity Specimen Wax Pattern.





Neg. No. G054370-3

Figure 2. Typical Vented Fluidity Wax Pattern.



Neg. No. G054370-5

Figure 3. Gating Arrangement of Combination Specimen Wax Pattern.



Neg. No. G054370-1

Figure 4. Vented Fluidity Specimen Casting - B-66 Alloy.

RADIOGRAPH

VISUAL

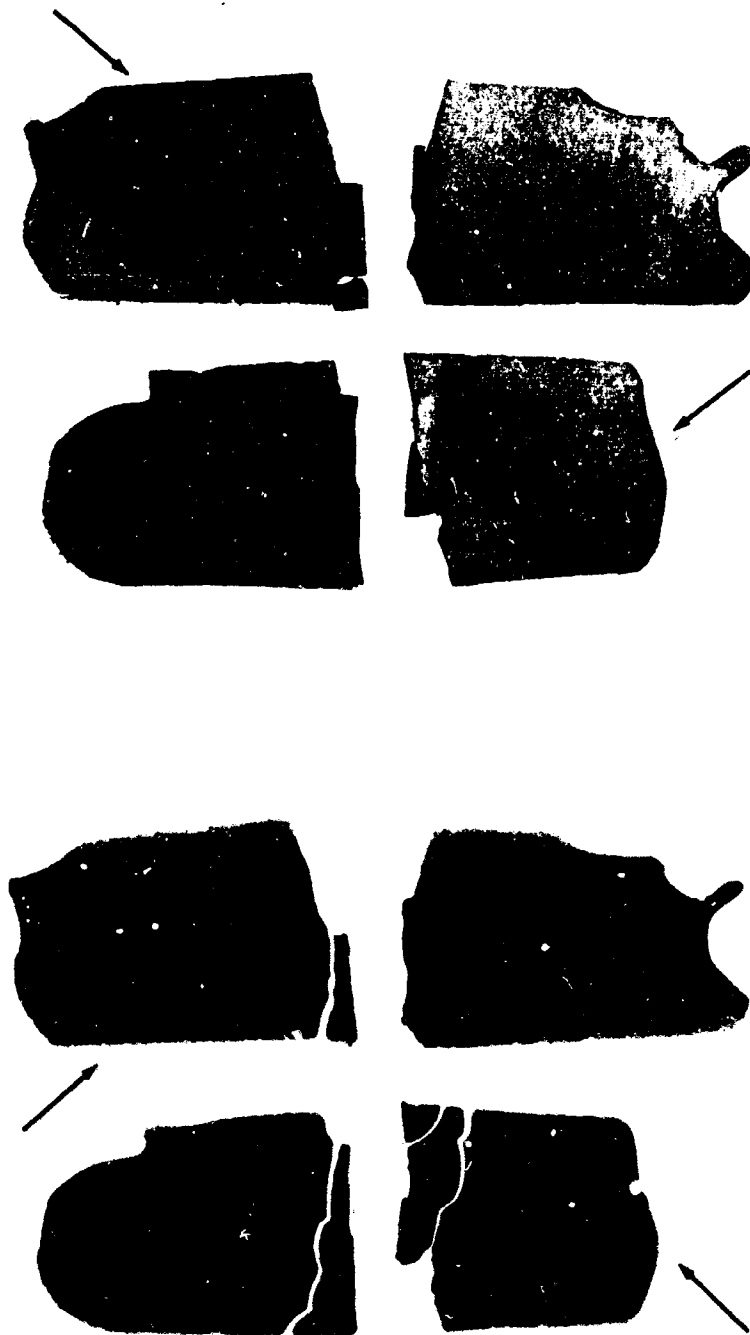
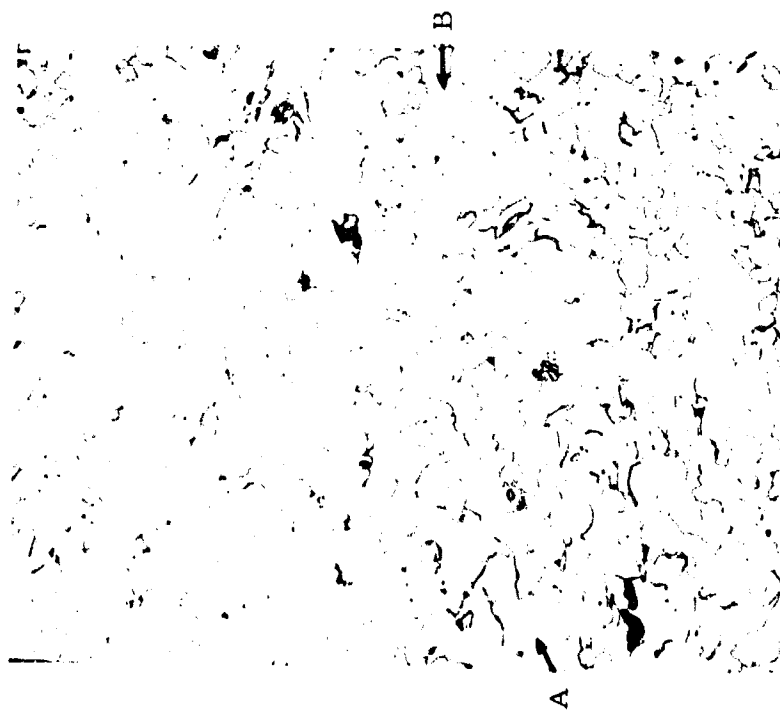
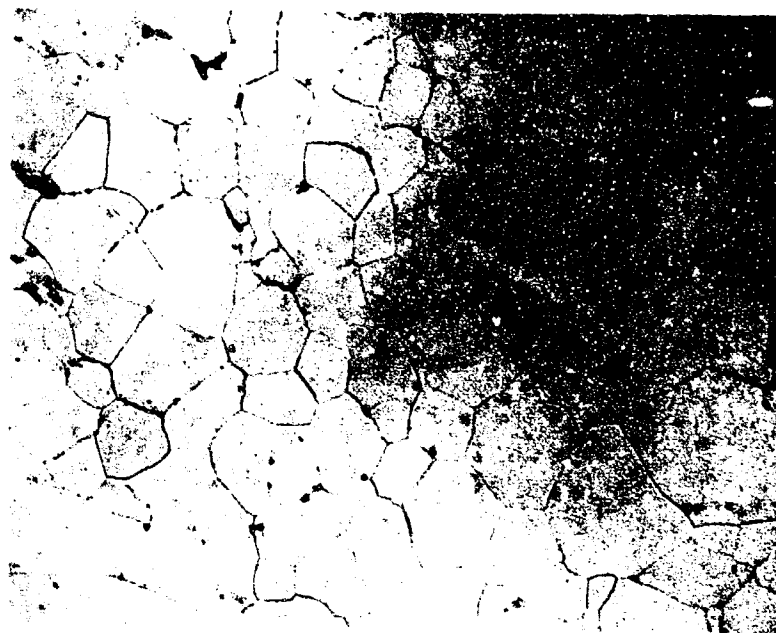


Figure 5. Radiographic and Visual Views of Cast B-66 Fluidity Wings - Vented (Arrows) and Non-Vented.

ABNORMAL



NORMAL



Neg. No. M70-1228

Mag. 100X

Neg. No. M70-0731

Mag. 100X

Etchant: 5 HNO<sub>3</sub>, 14 H<sub>2</sub>SO<sub>4</sub>, 20 HF, 50 H<sub>2</sub>O

Figure 6. Abnormal and Normal Microstructure - B-66 Alloy (Area A = Matrix, Area B = Suspected Contamination).



Neg. No. G049570-1

Figure 7. Vented Fluidity Specimen Casting, Fractured  
During Cleaning - C-3015 Alloy.

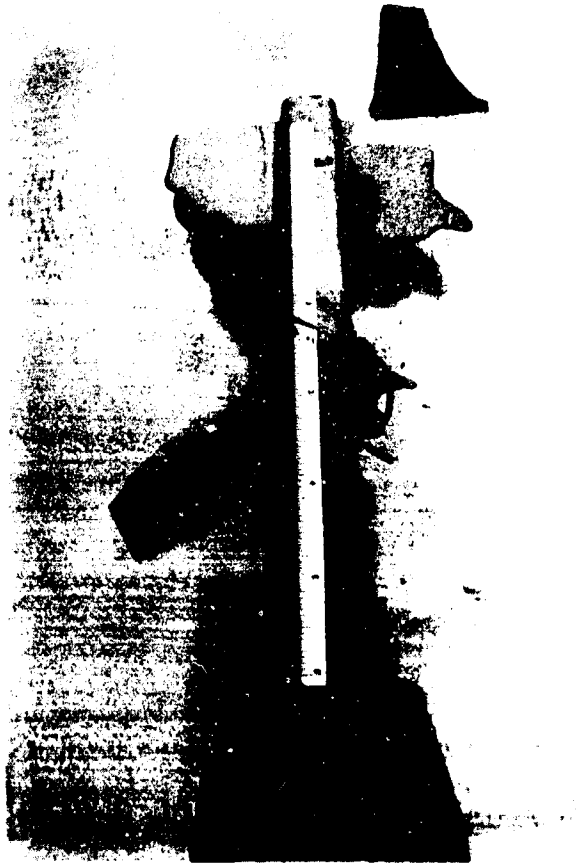


Neg. No. M70-1134

Mag. 100X

Etchant: Anodized

Figure 8. Internal Cracking in Segment of Vented Fluidity Specimen Casting - C-3015 Alloy.



Neg. No. G054370-4

Figure 9. Vented Fluidity Specimen Casting - C-3015 Alloy.



RADIOGRAPH

VISUAL

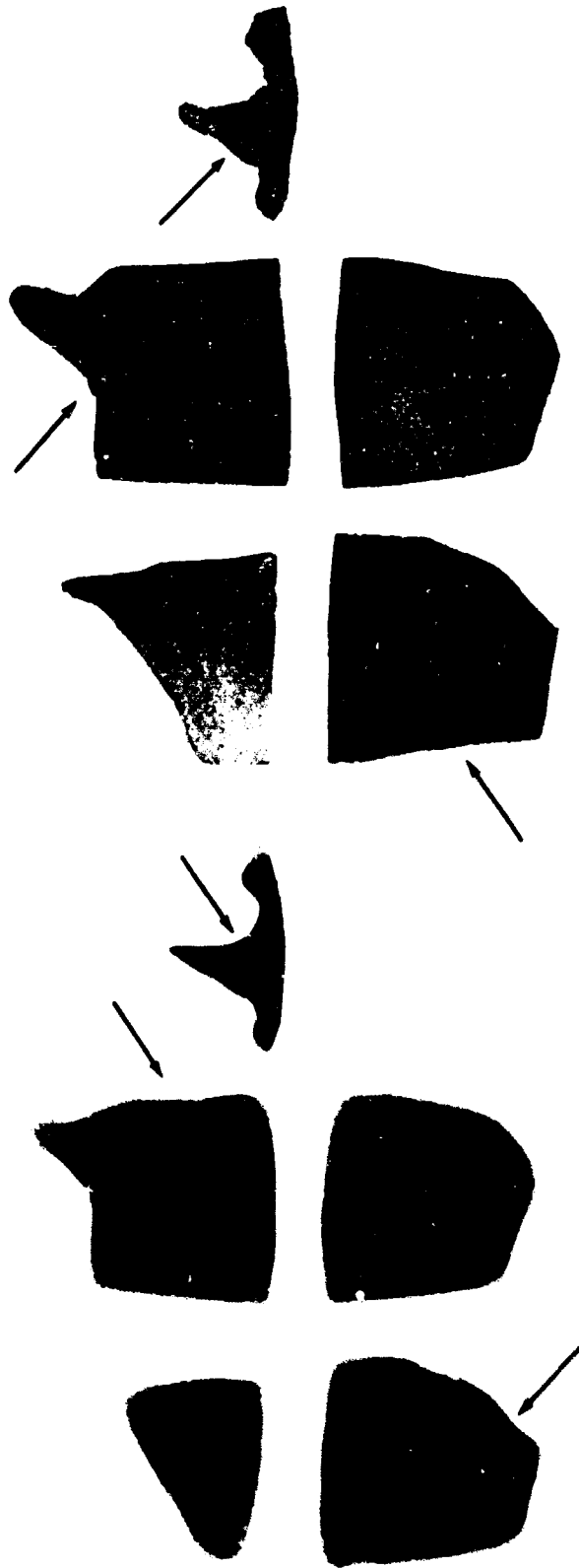
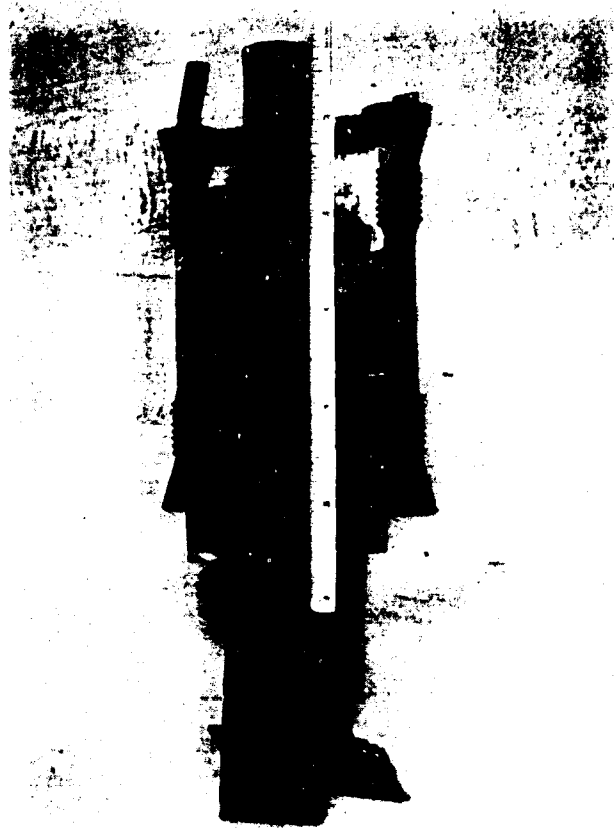


Figure 10. Radiographic and Visual Views of Cast C-3015 Fluidity Wings - Vented (Arrows) and Non-Vented.



Neg. No. G054370-2

Figure 11. Combination Specimen Casting - C-3015 Alloy.

RADIOGRAPH

VISUAL

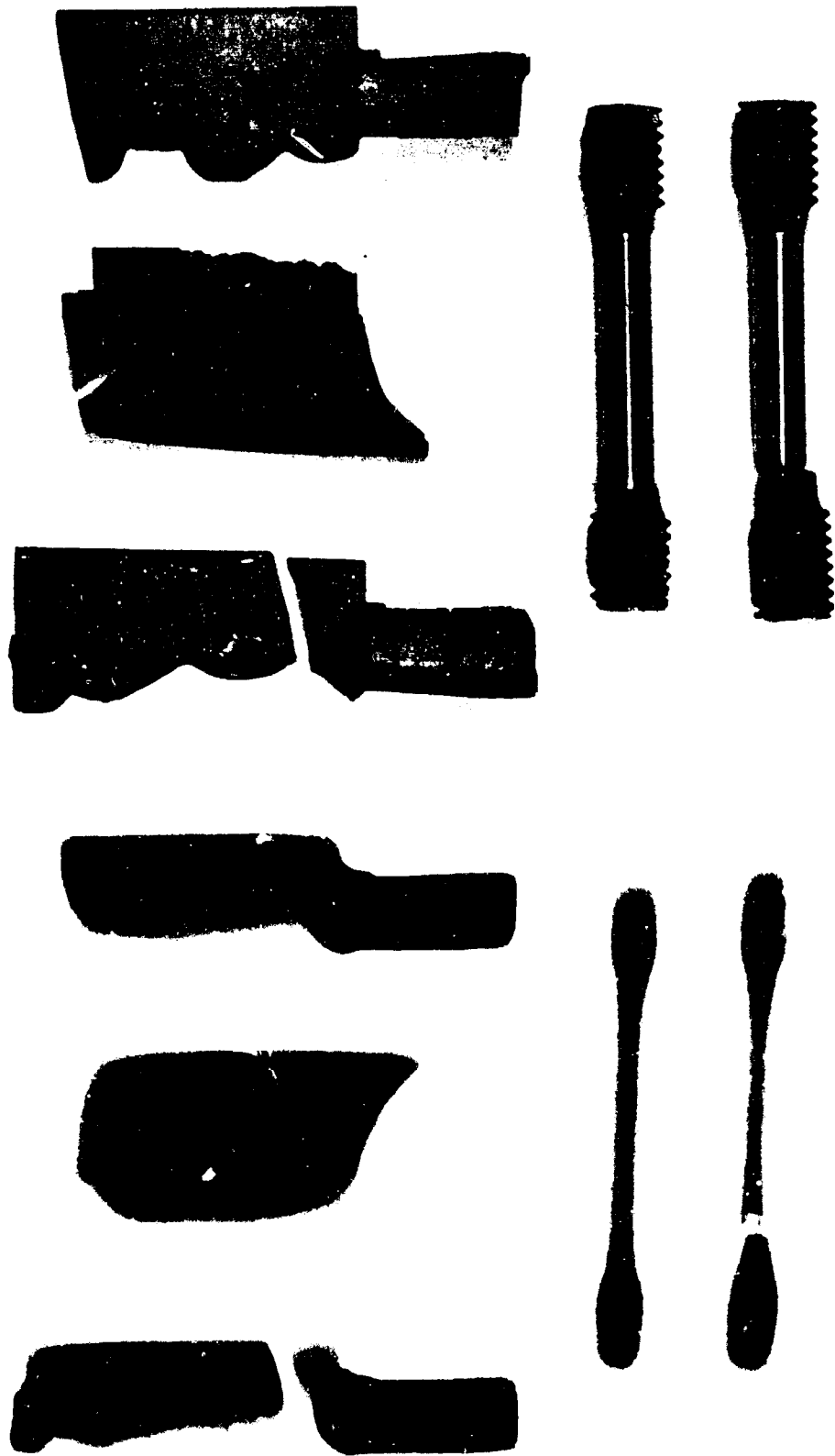
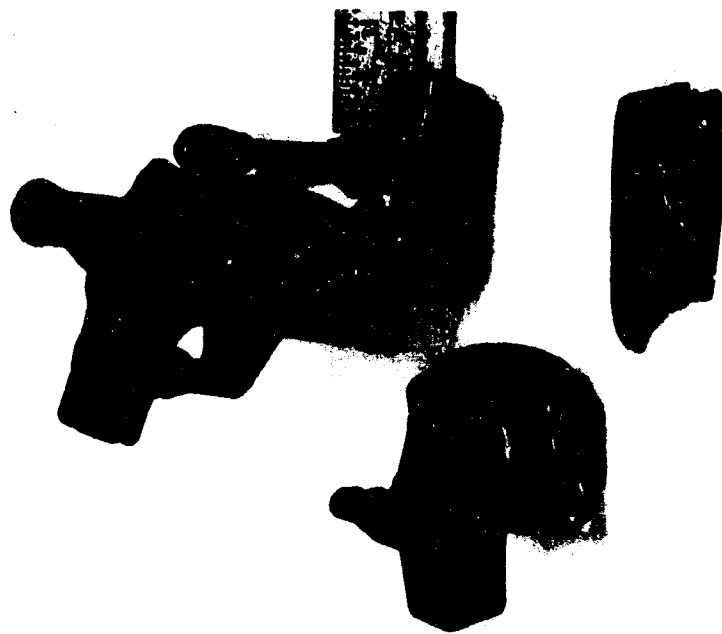


Figure 12. Radiographic and Visual Views of Cast C-3015 Test Specimens.



View A



View B

Figure 13. Modified Combination Specimen Casting - C-3015 Alloy.



Tungsten  
Interface

Zone A

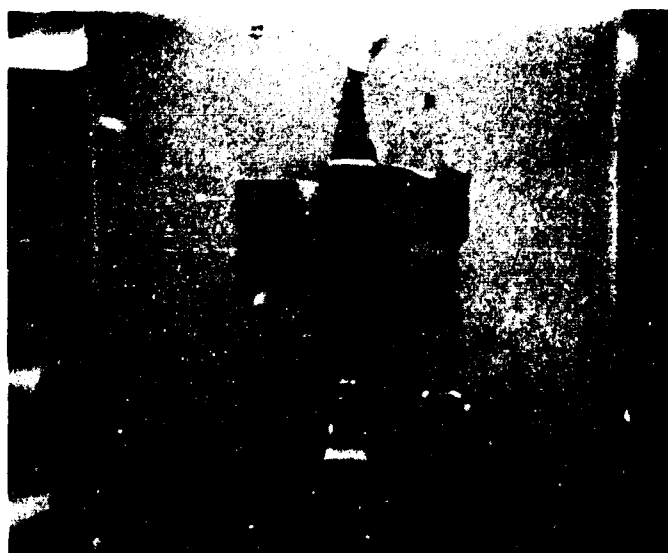
Oxidized  
Surface

Normal  
C-3015

Neg No. M70-1230  
Etchant: Anodized

Mag. 128X

Figure 14. Abnormal Microstructure - C-3015 Alloy.



Configuration A



Configuration B

Figure 15. Two Modified Gating Arrangements of Combination Specimen Wax Patterns.

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